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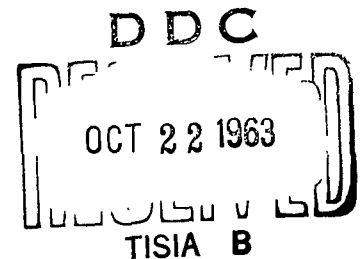
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Report RMD 5026-F
Contract No. NObs 88318
Index No. SF-007-03-03
Tasks 1000 and 1003

DIELECTRIC MATERIALS FOR HIGH TEMPERATURE APPLICATION

Report Period: 15 May 1963 to 1 September 1963

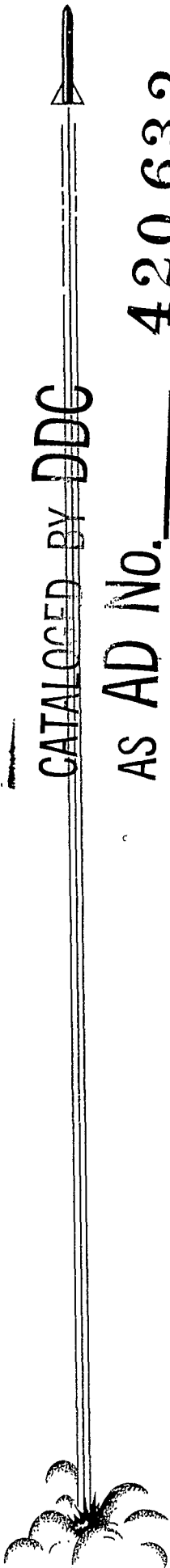


Thiokol®
CHEMICAL CORPORATION
REACTION MOTORS DIVISION
DENVER, NEW JERSEY

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DIELECTRIC MATERIALS FOR HIGH TEMPERATURE APPLICATION

Joseph Green
Nathan Mayes

Report Period: 15 May 1963 to
1 September 1963

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Tasks 1000 and 1003

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ABSTRACT

Research was conducted toward the preparation of thermally stable carborane silicone materials for use as embedding compounds and elastomers. End-blocked carborane silicones were prepared for use as plasticizers. Work was continued toward the preparation and isolation of cyclosiloxanes and carborane derivatives of cyclosiloxanes.

The electrical properties at high temperature and the thermal stability of the carborane silicone polymer were investigated. Fiber glass cloth laminates were prepared from the carborane silicone polymer and adhesion to metals was investigated.

ADMINISTRATIVE INFORMATION

Research efforts to prepare thermally stable dielectric materials and elastomers were originally authorized by Bureau of Ships Contract NObs 84774, 15 June 1961. The Navy Index Number was SR-007-03-03, Task 1000. That contract expired 15 December 1962. Technical work was resumed 15 February 1963 under Bureau of Ships Contract NObs 88318, Index Number SF-007-03-03, Tasks 1000 and 1003.

The Bureau of Ships Project Engineer is Mr. William Shetterly. The Project Supervisor at Thiokol Chemical Corporation, Reaction Motors Division is Mr. Joseph Green. Other contributing personnel include Mr. E. Cottrill, Mr. A. Jackson, Mr. A. Lum and Mr. N. Mayes.

SUMMARY

THE PROBLEM

The program objectives are to prepare carborane-based compounds useful as dielectric materials over the range of 25 to 500°C and prepare carborane-based elastomers useful over the same range. The immediate problem areas are:

- Defining structure-stability interrelationships
- Preparing high molecular weight linear polymer
- Conducting curing studies for application development.

FINDINGS

Area 1.

A carborane silicone polymer containing vinyl groups was prepared.

Linear carborane silicone polymer free of catalyst residue and free of cyclosiloxanes was prepared.

End-blocked, low molecular weight, liquid carborane siloxanes were prepared for use as high temperature plasticizers. Thermal stability of these materials is comparable to that of the polymer.

Area 2.

The resistance of mono-, di- and trihydrocyclotrisiloxane toward rearrangement was elucidated. The di- and trihydro materials are relatively unstable and may not be used to prepare carborane analogs.

Materials that are apparently carborane derivatives of mono- and dihydrocyclotetrasiloxanes were prepared.

Area 3.

Good quality fiber glass cloth laminates were fabricated from carborane silicone polymer. Tests of the adhesion of the polymer to metals showed good adhesion to copper, stainless steel, aluminum and tin.

I. BACKGROUND

Several years ago, Thiokol-RMD demonstrated the outstanding thermal stability of the carborane nucleus (Ref 1). On the basis of this work, a contract was awarded by the Bureau of Ships to Thiokol Chemical Corporation, Reaction Motors Division, for the preparation and evaluation of prototype carboranes as model compounds for ultrahigh temperature dielectric materials (Ref 2). The thermal stability and dielectric results obtained on the initial contract (Ref 3) were sufficiently promising so that a continuation contract was awarded in which the goals were:

1. To study and develop thermally stable carborane-based dielectric embedding materials.
2. To study and develop carborane-based elastomers for high temperature application.

The target characteristics for the embedding materials were:

- Should be suitable for embedding electronic parts capable of continuous operation at 350°C and 3000 hours at 500°C.
- Should have minimum shrinkage and be nonporous.
- Should closely match metals in thermal expansion and be capable of withstanding thermal shock.
- Should have the following electrical characteristics over the temperature range, ambient to 500°C.
 - Volume resistivity of 10^{10} ohm-cm (min)
 - Dissipation factor of 0.07 (max)
 - Dielectric constant of 5.0 (max) at frequencies of 1 mc and 100 mc
 - Dielectric strength of 1000 volts/mil at 60 cps.

- Should have a volume resistivity of 10^{13} ohm-cm (min) at 23°C after 230 hours at 70°C suspended over water in a sealed container.
- Should be self-extinguishing when tested as specified in Military Specification MIL-I-16823.

The required characteristics of the thermally stable elastomers were:

- Tensile strength of the vulcanized elastomer should be greater than 600 psi when tested at ambient temperature up to 500°C .
- Ultimate elongation of the vulcanized rubber should be not less than 200 percent when tested at 500°C .
- Shore A durometer hardness should not change more than 20 points after the elastomer has been subjected to oven aging at 500°C for 300 hours.
- Vulcanizates produced from the carborane gum elastomers should have the following initial properties:

Tensile Strength, min	1500 psi
Ultimate Elongation, min	300%
Hardness (Shore A)	between 40 and 80.

- Vulcanizates should not be affected appreciably by immersion in water at 212°F and should not absorb more than one percent of its weight in water when immersed for 30 days at $75 \pm 3^{\circ}\text{F}$.

Under the continuation contract (NObs 84774), a number of carborane-based materials were synthesized and evaluated for thermal stability and electrical properties. They included carborane-fluorocarbon polyesters, carborane-epoxies, carborane-phenylenes and carborane-silicones. Of these, the polyesters did not demonstrate the required thermal stability, and the epoxies and phenylene derivatives, although promising on the basis of thermal properties, presented difficulties in synthesis that removed them from consideration except as a long range research effort. The silicone derivatives showed excellent promise with electrical properties that met the program specifications and thermal stability at temperatures greater than 400°C . Work was therefore concentrated in this area, and the program was reoriented

into three phases: (1) improvement of the process for monomer and polymer synthesis to allow for early scaleup, (2) studies leading to higher molecular weight polymers for elastomer and embedding compound applications and (3) curing studies.

By the end of the contract period, significant progress had been made in each phase. Process development studies resulted in greatly improved yields and shorter reaction times. For example, starting with bromomethylcarborane, the overall yield of the monomer, (4-carboranylbutyl)methyldichlorosilane, was increased from 8 to 52 percent, and the preparative time was substantially reduced. Changing the method of polymer preparation from catalyzed condensation to cohydrolysis reduced polymerization time from five days to two hours. In the second area, polymer molecular weights of ca. 2000 obtained initially were increased to ca. 13,000, and progress was made in isolating cyclo-siloxanes through which polymers of much higher molecular weight may ultimately be prepared. Initial curing studies indicated that the liquid polymer could be cured through reactive end groups and showed that the carborane silicones could be cured in the several ways common in silicone polymer technology.

The present contract, which is an extension of the previous one, was awarded on the basis of previous results and the potential ability of the carborane silicones to meet the goals of the program. The goals and target requirements remain the same as does the orientation of the work of the program. Greater emphasis has been placed on the preparation of higher molecular weight polymer and polymer curing to obtain materials of suitable physical properties for initial applications evaluation.

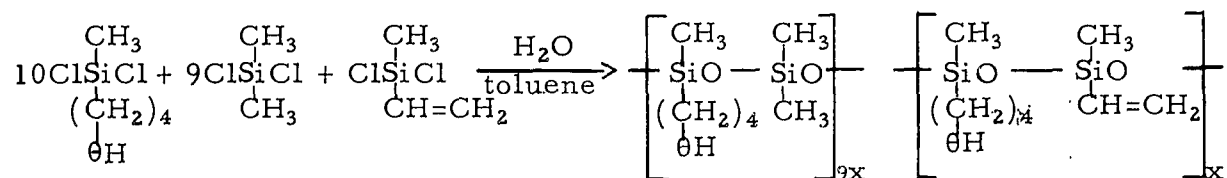
II. RESULTS

PHASE I. PREPARATION OF THERMALLY STABLE MATERIALS

A. Preparation of Carborane Siloxane Polymers Containing Curing Sites

The preparation of carborane silicone terpolymers which contain silyl hydrogen and/or silyl hydroxy groups to serve as curing sites have been reported (Ref 4). Additional quantities of these materials were prepared for use in fabrication of laminates and evaluation of adhesion to metals (cf. Section II, Phase I, E).

Another polymer containing functional groups was prepared for the first time during this report period. It is a terpolymer of (4-carboranylbutyl)methyldichlorosilane, dimethyldichlorosilane and methylvinylidichlorosilane in the molar ratio of 10:9:1.



Polymerization was accomplished by cohydrolysis of the silanes in a water-toluene mixture at room temperature. The product was obtained as a viscous liquid very similar to other carborane silicone polymers prepared using the same hydrolysis procedure.

B. Preparation of "Alternating" Carborane Siloxane Polymer

The polymerization of 1,1,3-trimethyl-3-(4-carboranylbutyl)-1,3-dichlorodisiloxane to a copolymer with a certain degree of structural regularity has been reported (Ref 4). Since no improved thermal stability was noted, further work in this area was not anticipated. It has now been shown, however, that this material may be hydrolyzed to produce linear polymer of sufficiently high molecular weight (greater than 4000) to allow separation from cyclic

products by solvent fractionation. Heretofore, it was necessary to use catalysts such as tin octoate or dibutyl tin dilaurate for this purpose. The catalyst residues in the polymer apparently caused decomposition at higher temperatures. The higher molecular weight linear polymer, catalyst free, and freed of cyclics by solvent fractionation may be used to determine if, and at what temperature, thermal rearrangement of linear to cyclic occurs. The preparation of an additional quantity of this polymer to be used in thermal rearrangement studies has been initiated with the preparation of 1,1,3-trimethyl-1,3-dichlorodisiloxane as previously described (Ref 4). Approximately 70 g of material has been obtained.

C. Fabrication and Curing Studies

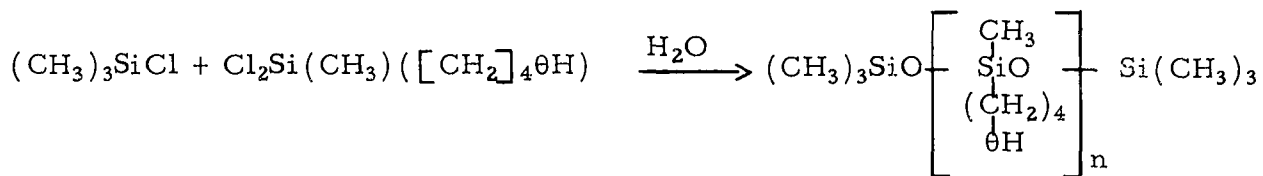
A brief review of methods for curing siloxane polymers and the curing of carborane silicone materials by some of these methods (reaction through the functional groups SiH and SiOH) has been presented (Ref 4). Further curing studies have been done to evaluate free radical curing systems. Carborane silicone was treated with benzoyl peroxide in attempts to cure through reaction of SiCH₃ groups. There was no evidence of curing in several attempts carried out under conditions employed for curing dimethylsilicone. Another free radical cure was attempted on a polymer that contained 5 molar percent vinyl silane units. Again conditions were employed under which a corresponding noncarborane polymer may be cured, di-*tert*-butyl peroxide at a temperature of 170°C. There was some curing as shown by the change from liquid to a soft rubber while hot and to a brittle solid when cooled. The material was no longer completely soluble in acetone but formed a soft gel. Prolonged extraction with acetone, however, resulted in almost complete extraction and indicated a very low state of cure.

The carborane silicones that could be cured through SiOH and SiH groups were used in the fabrication of fiber glass cloth laminates. Laminates were prepared by impregnating fiber glass cloth with the uncured polymer containing catalyst (either tin octoate or dibutyl tin dilaurate), then partially curing the silicone in place by the application of heat. The cloth was then stacked in a mold and press-cured with heat and pressure. The SiOH polymer, cured with tin octoate, was successfully used in this process to produce high quality laminates. The SiH polymer laminates were slightly tacky, a result that was attributed to insufficient crosslinking. A number of laminate samples were prepared and the applicability of carborane silicone for laminating was amply demonstrated.

The potential of carborane silicone for coating applications was evaluated by curing SiOH polymers in place on metal test panels. This was done by casting films of the polymer containing tin octoate catalyst onto stainless steel, aluminum copper and tin test panels. Films of 5 to 10 mil thickness and others of 1 mm thickness were then cured at 120°C. In every case the clear transparent films adhered well to the metal surfaces and the panels could be flexed many times without cracking or peeling of the resin.

D. Carborane-Siloxane Plasticizers

In anticipation of the need of a high temperature plasticizer to be used in fabrication of molded or milled items, low molecular weight end-blocked carborane siloxanes were synthesized. The compounds were made by cohydrolysis of trimethylchlorosilane and (4-carboranylbutyl)methyldichlorosilane. This procedure resulted in a mixture consisting of trimethylsiloxy terminated polysiloxanes of varying molecular weight.



where $n = 1, 2, 3, \dots$

The molecular weight distribution was dependent upon the molar ratio of the reactants. Thus when an equimolar ratio was employed, a high proportion of the product was of high molecular weight and could not be distilled. The use of a large excess of trimethylchlorosilane resulted in a large proportion of product where $n = 1$ (I) and smaller proportions of product where $n = 2$ (II) and 3 (III). Polysiloxanes I, II and III were isolated by a "short path" distillation. I was collected at 133 to 142°C/0.0035 mm Hg as a water white, mobile liquid (n_D^{25} , 1.4782). The infrared spectrum supported the proposed structure.

Analysis for $\text{C}_{13}\text{H}_{40}\text{O}_2\text{B}_{10}\text{Si}_3$, Calculated: B, 25.7; Mol Wt, 421
Found: B, 22.4; Mol Wt, 470

II was collected at 260°C/0.004 mm Hg as a slightly cloudy liquid, (n_D^{25} , 1.5047).

Analysis for $\text{C}_{20}\text{H}_{62}\text{O}_3\text{B}_{20}\text{Si}_4$, Calculated: B, 31.8; Mol Wt, 679
Found: B, 33.8; Mol Wt, 710

III distilled at an undetermined temperature and only a few drops were collected; however, it was necessary to heat the distilling flask at well over 400°C to effect distillation. A very viscous cloudy liquid was obtained (n_D^{25} , 1.5159).

Analysis for $C_{27}H_{84}O_4B_{30}Si_5$, Calculated: B, 34.6

Found: B, 35.5

E. Higher Molecular Weight Polymers Through Cyclosiloxanes

The isolation of a material believed to be bis(4-carboranylbutyl)hexamethylcyclotetrasiloxane was accomplished by solvent fractionation and molecular distillation of (4-carboranylbutyl)methyldichlorosilane/dimethyldichlorosilane hydrolyzate and has been reported (Ref 4). Also reported was the preparation of some hydrocyclosiloxanes to be used in a more direct synthesis of carborane cyclosiloxanes by addition of butenylcarborane. Cyclotri- and cyclotetrasiloxanes were prepared and obtained as a multi-component mixture which was subjected to fractional distillation.

It was observed that the cyclotrisiloxanes were apparently unstable and equilibrium occurred with the formation of higher boiling products. This phenomenon was investigated with the aid of gas chromatography and it was observed that in a mixture containing trimethyl-, tetramethyl-, pentamethyl- and hexamethylcyclosiloxanes, the trimethyl variety disappeared within one hour at room temperature. The tetramethyl analog was more stable and a small portion remained after one week at room temperature. The concentration of the pentamethyl and hexamethyl derivatives remained practically constant over the one week period. The major product of the cyclotrisiloxane rearrangements was hexamethylcyclotetrasiloxane.

Carborane cyclosiloxane derivatives were prepared by the reaction of butenylcarborane with a distillate fraction believed to be hexamethylcyclotetrasiloxane. The distillate fraction was not pure, however, and at least two carborane derivatives were obtained. Molecular distillation of the reaction products yielded a material at 155 to 160°C/0.004 mm Hg which crystallized to a waxy solid of mp 35 to 42°C.

Analysis for $C_{13}H_{40}O_4B_{10}Si_4$, Calculated: B, 22.50

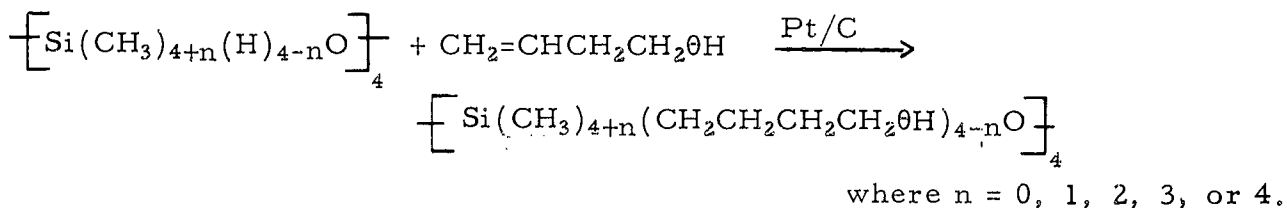
Found: B, 22.55

Another material was obtained at 275 to 280°C/0.004 mm Hg; it crystallized to a solid of mp 85 to 110°C.

Analysis for $C_{18}H_{56}O_4B_{20}Si_4$, Calculated: B, 32.27

Found: B, 31.74

The products are believed to be (4-carboranylbutyl)heptamethylcyclotetrasiloxane and bis(4-carboranylbutyl)hexamethylcyclotetrasiloxane, respectively, formed as illustrated in the equation below:



The wide melting range of the materials indicated that impurities were present. Efforts to purify by recrystallization were unsuccessful; the materials were very soluble in all the organic solvents that were tried. Subjection of the samples to elution chromatography resulted in the isolation of a crystalline solid of mp 46 to 47°C from the lower fraction, but no substantial reduction in the melting range of the higher fraction could be obtained. The higher fraction may be a mixture of the isomers 1,5-bis(carboranylbutyl)hexamethylcyclotetrasiloxane and 1,3-bis-(carboranylbutyl)hexamethylcyclotetrasiloxane. Proof of structure of the purified material awaits mass spectrographic and elemental analyses. The higher fraction is being subjected to gas chromatographic and mass spectrographic analyses in efforts to purify and identify.

PHASE II. THERMAL EVALUATION

The results of TGA and "closed tube" thermal stability measurements of carborane silicone polymers of differing structure and method of preparation have been reported (Ref 4). The "closed tube" results were inconclusive since the polymer samples were found to contain traces of solvent. These were repeated after rigorous treatment to remove any volatile impurities. The samples rechecked were those prepared by: (1) ferric chloride catalyzed condensation of (3-methylcarboranylpropyl)methyldichlorosilane and dimethyldiethoxysilane, (2) the ferric chloride catalyzed condensation of (4-carboranylbutyl)methyldichlorosilane and dimethyldiethoxysilane, (3) the ferric chloride catalyzed condensation of (4-carboranylbutyl)methyldiethoxysilane and dimethyldichlorosilane, (4) the cohydrolysis of (4-carboranylbutyl)methyldiethoxysilane and dimethyldiethoxysilane and (5) chain extension of (3) by tin octoate catalyzed condensation of hydroxy terminals. The samples were freed of volatiles by heating at 200°C/0.1 mm Hg for 3 hours. The samples,

sealed in evacuated Pyrex glass tubes, were treated at 350°C for periods up to 360 hours. There was a small pressure increase denoting some decomposition for each sample except (3). This material showed no pressure increase after 233 hours at 350°C. However, this result could not be duplicated in repetitions of the measurement on the same sample and on other samples of the same material.

The thermal stability of material prepared for use as plasticizer, (cf. Phase I, C), was evaluated by "closed tube" measurements at 350°C. There was no visible pressure increase after 24 hours. After 89 hours and 163 hours, pressure increases corresponding to a rate of 5×10^{-5} moles gas/g-hr were observed. If the average molecular weight of the decomposition gases is 16, then this represents an initial decomposition rate of 0.08%/hour.

Evaluation of electrical properties over the temperature range 25 to 375°C was done for one of the uncured carborane silicone polymers, (3). The material was tested at the Brooklyn Navy Yard for dissipation factor, dielectric constant and volume resistivity. Results of the measurements indicated areas for possible improvement. The dissipation factor and dielectric constant were comparatively high, the insulation resistance relatively low and the dielectric constant unusually variable. It is noted that much of the variability in dissipation factor and dielectric constant in the 50 to 100°C temperature range is due to the change in state from solid to liquid. Further, catalyst residues and impurities probably contribute to these properties. The polymer lost no weight during this evaluation; however, it developed an amber color. It was returned to Thiokol-RMD and the amber color removed by passing a benzene solution through a silica column to yield a clear, slightly yellow polymer.

III. CONCLUSIONS AND RECOMMENDATIONS

A. ACCOMPLISHMENTS AND CONCLUSIONS

Carborane silicone polymer could not be cured with benzoyl peroxide which is not consistent with dimethylsilicones. This suggests that carborane may sterically hinder attack at the methyl group. Such behavior further suggests that carborane may increase stability to oxidation by suppressing free radicals and appears to merit investigation at some future date.

The carborane silicone polymer of greater than 4000 molecular weight, prepared without the use of catalysts, can be used to study thermal rearrangements of carborane silicones. This particular material is useful because it has no catalyst residues present and because it has been fractionated to remove low molecular weight materials including cyclics. The material may be subjected to various high temperatures, and any equilibration of linear to cyclic may be determined by changes in the number average molecular weight of the treated material. Since the material is initially cyclic free, the formation of cyclics may also be determined by mass spectrometry.

It is necessary to study possible rearrangement in this manner because of the high boiling temperatures of carborane cyclosiloxanes. In the case of dimethylsiloxanes the equilibration temperature may be determined by observing the temperature at which volatile cyclic products appear and begin to distill. The carborane cyclosiloxanes boil at temperatures in excess of the decomposition temperature (the lowest boiling carborane cyclotetrasiloxane boils at 155 to 160°C/0.004 mm Hg. this extrapolates to ca. 450°C/760 mm Hg) and must be detected by other means.

It is believed that very high molecular weight carborane silicone polymers must be prepared by ring opening procedures analogous to dimethylsilicone. Toward this end, two carborane cyclotetrasiloxanes have been prepared and the feasibility of preparing a carborane cyclotrisiloxane has been shown. One of the carborane cyclotetrasiloxanes may actually be a mixture of two isomers and separation of these by gas chromatography should be investigated.

The practical development of carborane silicone polymer was pursued with the fabrication of fiber glass laminates and demonstrations of the applicability of the material for use in coating metals. In both areas the material was shown to have good properties and to warrant further investigation and development.

B. RECOMMENDATIONS

The following work outlined is recommended for the continued program:

- Prepare larger quantities of polymer for development studies.
- Prepare a carborane cyclotrisiloxane.
- Study ring opening polymerization of carborane cyclosiloxanes to yield high molecular weight polymer.
- Study thermal rearrangements in carborane silicone polymer.

IV. REFERENCES

1. Report RMD 1147-E1, Development of Solid Propellants for Application in Extended Temperatures or Space Environment Conditions, Thiokol Chemical Corporation, Reaction Motors Division, Contract AF33(616)-6424.
2. Contract NObs-78416, Index No. SR-007.
3. Report RMD 2034-F, Ultra High Temperature Dielectric Materials, Thiokol Chemical Corporation, Reaction Motors Division, Contract NObs-78416.
4. Report RMD 5026-Q1, Dielectric Materials for High Temperature Application, Thiokol Chemical Corporation, Reaction Motors Division, Denville, New Jersey, Contract NObs-88318.

<p>THIokol CHEMICAL CORPORATION, REACTION MOTORS DIVISION, DENVILLE, N. J., DIELECTRIC MATERIALS FOR HIGH TEMPERATURE APPLICATION, by Joseph Green and Nathan Mayes, September 1963, 18 p. (Index No. SF-007-03-03, Tasks 1000 and 1003, Contract No. NObs 88318).</p> <p>Research was conducted toward the preparation of thermally stable carborane silicone materials for use as embedding compounds and elastomers. End-blocked carborane siloxanes were prepared for use as plasticizers. Work was continued toward the preparation and isolation of cyclosiloxanes and carborane derivatives of cyclosiloxanes.</p> <p>The electrical properties at high temperature and the thermal stability of the carborane silicone polymer were investigated. Fiber glass cloth laminates were prepared from the carborane silicone polymer and adhesion to metals was investigated.</p>	<p>UNCLASSIFIED</p> <ol style="list-style-type: none"> 1. Preparation of Thermally Stable Materials 2. Thermal Evaluation <p>I. Contract NObs 88318</p>	<p>THIokol CHEMICAL CORPORATION, REACTION MOTORS DIVISION, DENVILLE, N. J., DIELECTRIC MATERIALS FOR HIGH TEMPERATURE APPLICATION, by Joseph Green and Nathan Mayes, September 1963, 18 p. (Index No. SF-007-03-03, Tasks 1000 and 1003, Contract No. NObs 88318).</p> <p>Research was conducted toward the preparation of thermally stable carborane silicone materials for use as embedding compounds and elastomers. End-blocked carborane siloxanes were prepared for use as plasticizers. Work was continued toward the preparation and isolation of cyclosiloxanes and carborane derivatives of cyclosiloxanes.</p> <p>The electrical properties at high temperature and the thermal stability of the carborane silicone polymer were investigated. Fiber glass cloth laminates were prepared from the carborane silicone polymer and adhesion to metals was investigated.</p>	<p>UNCLASSIFIED</p> <ol style="list-style-type: none"> 1. Preparation of Thermally Stable Materials 2. Thermal Evaluation <p>I. Contract NObs 88318</p>
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